ISOTOPIC ANALYSIS OF FISSION-PRODUCT IODINE-131

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Submitted in partial fulfillment of
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IN
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ABSTRACT

The presence of isotopic impurities in radioactive preparations may be determined by neutron activation analysis. The I-127 present in carrier-free I-131 was redetermined. The results indicate the presence of 0.7 milligrams of I-127 per lambda of I-131 solution which assayed 30.4 microcuries per lambda. This is equivalent to 1.8×10^{19} atoms of I-127 per millicurie of I-131, about three times that reported by Tyler and Gminder [1] for a similar material. Activation of this material for 2.5 half-lives of I-130 at the same flux failed to show the presence of I-129.

The writers wish to express their appreciation for the invaluable assistance of Professor William W. Hawes of the U. S. Naval Postgraduate School, whose countless hours of assistance and limitless patience were extended during this investigation.

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1. INTRODUCTION.

Iodine-127 and I-129 are formed together with I-131 in fission products and are both present in the usual preparations of the latter isotope. Previous investigations of carrier-free I-131 using neutron activation techniques by Tyler and Gminder [1] have indicated the presence of relatively large quantities of I-127. This study, a continuation of previous work in determining the feasibility of applying activation analysis to the detection of isotopic impurities in radioactive preparations, had two specific purposes. First, to confirm the reported high I-127 concentration, and second, to extend the activation period in order to obtain, if possible, measurable quantities of I-130 for estimation of the I-129 present.

Since I-131 is obtained by a carrier-free separation of fission products, only those isotopes produced in the fission reaction should be present initially. Those isotopes which remain after a moderate decay period are: inactive I-127, I-129 (1.72 x 10^7 years), I-131 (8.08 days) and I-133 (20.8 hours) [3]. The composition of carrier-free I-131 is reported as greater than 99% I-131 and less than 1% I-133. No values for I-127 and I-129 are given. The literature indicates that in 60-day irradiated slugs for I-131 preparation, significant amounts of I-127 and I-129 are formed. [4,5]

If I-127 and I-129 are to be determined by activation, the thermal neutron cross sections for activation must be large enough for adequate amounts of I-128 and I-130 to be formed for detection. The energies of the gamma rays to be measured must be sufficiently removed from possible interfering emissions to permit resolution by the spectrometer. I-127 has a thermal neutron cross section of 5.5 ± 0.5 barns and I-129 has a cross section of 11 ± 4 barns. [6] The principle gamma radiation of I-128

lies at 0.45 mev and occurs in 17% of the decay. Similarly the most prominent gamma radiation of I-130 lies at 0.660 mev and may be observed in about 90% of the decay. The I-132 produced from I-131 is negligible at the flux employed.

2. EXPERIMENTAL DETERMINATIONS.

The material used was carrier-free I-131 obtained from Oak Ridge National Laboratory. It assayed 30.4 millicuries per milliliter and was allowed to decay for 43 days to an activity of 0.7 millicurie per milliliter. This activity permitted the use of samples as large as 5.0 lambda.

Samples were irradiated in the AGN-201 Reactor at the reactor core center position where the thermal neutron flux is rated at 4.5×10^6 neutrons per square centimeter per second. Irradiation times were 125 minutes for the I-127 determinations and 31 hours for the I-129 determinations corresponding to 5.0 and 2.5 product half-lives respectively.

Sample holders were machined of lucite and were cylindrical, 19 mm in inside diameter and 2 mm inside height. The sample of 5.0 lambda volume was pipetted, spread and evaporated to dryness. A 2 mm-thick cover was sealed in place. The absorption due to the cover was determined directly and found to be 0.02.

Gamma radiation rate was measured with a Tracerlab RLP-6 Stepwise Scanning Spectrometer which sequentially scanned 50 channels of energy. Input was from a 1 x $1\frac{1}{2}$ inch sodium iodide (thallium activated) scintillation crystal to a linear amplifier whose output was introduced into a discriminator prior to scanning and counting. Settings other than 100% spread and zero discriminator bias were found to introduce some distortions in spectral shape. No spread or discriminator bias was used.

The absolute gamma rate R is obtained from the measured gamma rate R_p by the expression

$$R = \frac{R_p}{p \quad e_t \quad A} = 132 \; R_p$$

in which the remaining quantities are p, the peak-to-total ratio for the

photopeak energy involved; e_t, the absolute crystal detection efficiency and A, the absorption of the intervening materials. Values for these factors are: for p, I-128 0.282; for e_t, 0.0274 and for A, 0.98. Data given by Heath [7] on the efficiency of scintillation crystals for disc sources was used to compute the absolute crystal detection efficiency. Peak-to-total ratios were determined for Au-198, Cs-137 and Zn-65. The value of 0.282 for the 0.45 mev gamma of I-128 was interpolated from these data.

Four samples were used in the I-127 determinations. These were identified with the numerals I through IV. Each sample was counted four times, runs 1 and 2 being those including the I-128; runs 3 and 4 were obtained after the I-128 had decayed below measurable levels. The latter are due to I-131 only. The data are presented in columns 1, 2 and 3, pages 11 through 26, Appendix I for the I-127 determination and pages 28 through 30 of Appendix II for I-129.

As shown in section 3 below, very large quantities of I-127 are indicated by the activation results obtained. The quantities are such that gravimetric chemical analysis should have some significance. 400 lambda portions of the material were examined. Precipitation with silver ion was negative, but after reduction according to the method of Glendenin and Metcalf [2] significant silver iodide precipitate was obtained. This analysis gave 0.1 milligrams of iodine per lambda which may now be attributed entirely to iodine in higher oxidation states.

3. RESULTS.

Each pair of runs that include I-128 were made at the same elapsed time following irradiation. The time interval between consecutive measurements was only 20 minutes, hence it is possible with sufficient accuracy to decay all I-131 data to the mean elapsed time of the I-128 determinations.

While the spectrometer is normally sufficiently stable in short-time drift the long-time drift is great enough that individual spectral determinations need to be normalized for comparison. In order to accomplish this the I-131 peaks at 0.364 mev and 0.638 mev were employed and the pulse height scale of individual determinations adjusted so that the positions of these peaks coincided. Moreover, since it was impractical to adjust the quantity of I-131 exactly, normalization in count rate is also necessary. All data were normalized arbitrarily to conform with that of run I-3.

All data for I-131 alone are shown graphically in Figure 1. This curve was subtracted point by point from those data that include I-128. This normalized data is recorded in columns 4 and 5, pages 11 through 26 of Appendix I. Following the method of Heath [8] the peak count rate and standard deviation for all data were determined assuming a gaussian distribution.

The resolution of the detector was such that these data were significant for a three-channel spread. A standard deviation appropriate to the resolution and energy involved was estimated. The differences were weighted according to the above estimate and a peak value at channel 14.73 (corresponding to an energy of 0.45 mev). The differences and their weighted values are recorded in columns 3 and 4, page 27, Appendix I. The weight-

COMPOSITE GRAPH OF I-131

SPECTRAL SHAPE

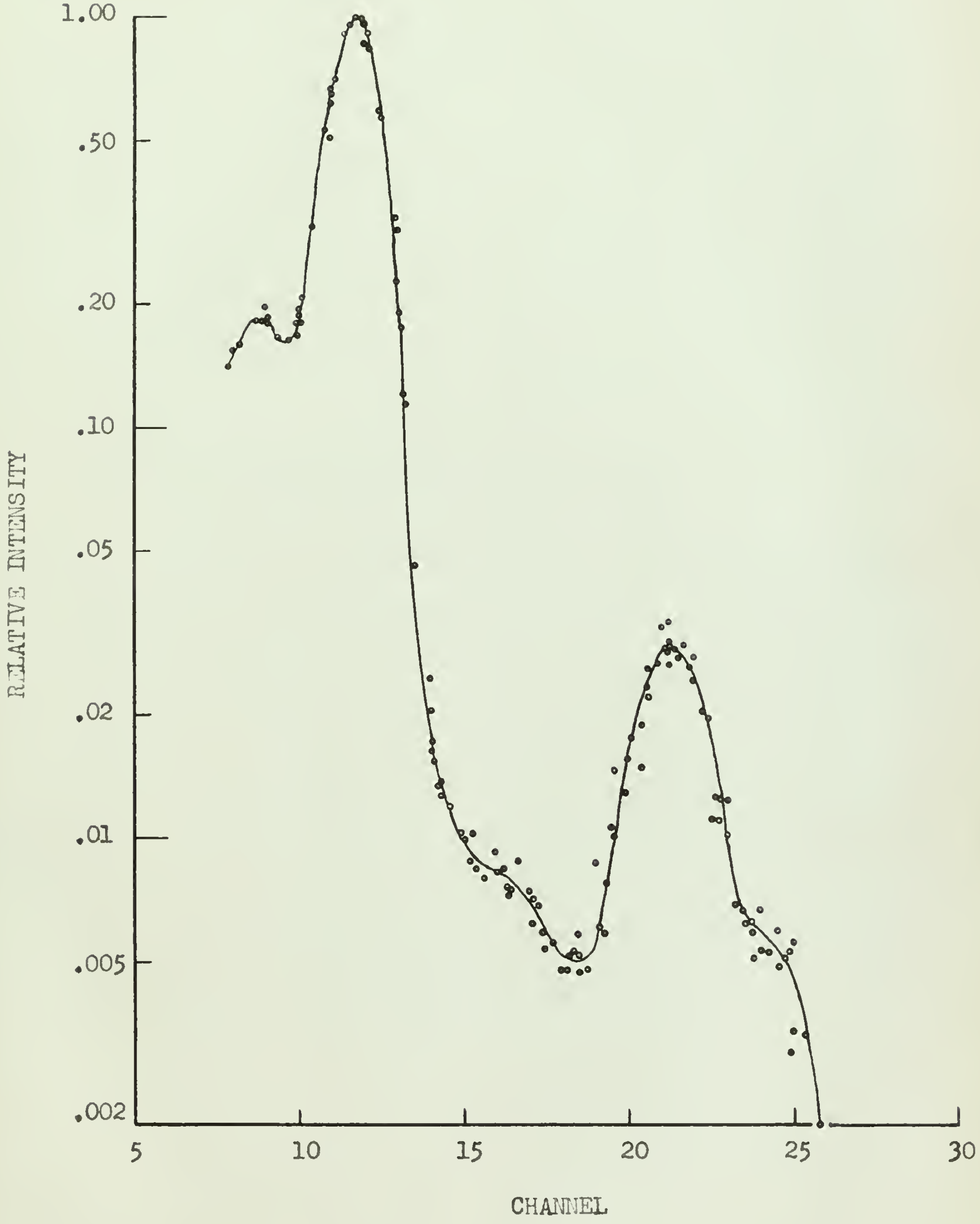

Figure 1

ed values of the differences were averaged with appropriate decay correction to give a peak height of 21.1 counts per minute for the I-128 peak.

From this value of peak height the integral count rate is determined and converted to absolute count rate by the equation previously given [8,9]. The average value obtained for integrated count rate is 33.4 counts per minute. The absolute count rate is 4,400 counts per minute. The activity is then given by dividing the absolute count rate by 0.17, the fraction of decay resulting in 0.45 mev gamma radiation. The quantity of I-127 is given by

$$\alpha = N \sigma \phi (1 - e^{-\lambda t})$$

or

$$N = \frac{26{,}000}{5.5 \times 10^{-24} \times 4.5 \times 10^{6} \times 60 \times .968}$$

This yields a final value of 1.8×10^{19} -20 atoms for a 5.0 lambda sample. This is equivalent to 0.76 milligrams per lambda or 1.2×10^{20} atoms per millicurie. In terms of relative amounts this represents a concentration of I-127 3×10^{6} times the concentration of I-131.

The data for the determination of I-129 was analyzed similarly. In the region of the 0.660 mev energy the data were found to coincide with the values determined for I-131 within the limits of statistical variation. It is evident that within the precision of these measurements no detectable amount of activity was present attributable to I-130.

4. CONCLUSIONS.

While some I-127 in separated fission-product I-131 is inevitable [4,5], the quantity found in the samples is much greater than one would expect. While great accuracy can not be claimed due primarily to limitations of neutron flux available for activation, the high result obtained has been confirmed in order of magnitude. Gravimetric determination of iodine gave a quantity about one-eighth as great. Further confirmation was obtained by irradiating a weighed amount of potassium iodide containing a quantity of iodine that was determined to be approximately three times the amount indicated by activation. Analysis of the data by the procedure used here gave a value 1.6 times the amount present. The discrepancy may be due to uncertainty in the neutron flux.

5. SUMMARY.

(1) Isotopic analysis by activation and gamma spectrum analysis offers promise for the determination of the composition of active materials.

(2) Iodine-127 found in carrier-free I-131 preparation amounts to 3.0×10^6 times the concentration of I-131.

(3) Iodine-129 was not detected under experimental conditions where it should have appeared if present in a quantity comparable with I-127.

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APPENDIX I

I-127 DETERMINATION

I-128 + I-131 RUN I-1

Channel	Obs. Count Rate (c/m)	Elapsed Time, min	Norm. Channel	Norm. Count Rate
8	5,575	6		
9	5,263	7		
10	10,195	9		
11	27,893	10		
12	21,854	11	12.43	.6829
13	2,527	12	13.51	.0790
14	400	13	14.59	.0125
15	287	15	15.67	.0090
16	221	16	16.75	.0069
17	134	17	17.83	.0042
18	188	18	18.91	.0059
19	418	19	19.88	.0131
20	930	21		
21	722	22		
22	263	23		
23	192	24		
24	139	25		
25	34	27		

I-128 + I-131 RUN I-2

Channel	Obs. Count Rate (c/m)	Elapsed Time, min	Norm. Channel	Norm. Count Rate
8	5,647	28		
9	5,281	29		
10	10,680	30		
11	30,695	32		
12	15,165	33	12.64	.4892
13	1,385	34	13.63	.0447
14	364	35	14.63	.0117
15	296	37	15.62	.0095
16	267	38	16.61	.0086
17	192	39	17.60	.0062
18	173	40	18.60	.0056
19	412	41		
20	801	42		
21	897	44		
22	441	45		
23	203	46		
24	167	47		
25	109	49		

I-131 Alone RUN I-3

Channel	Obs. Count Rate (c/m)	Elapsed Time, min	Norm. Channel	Norm. Count Rate
8	4,376		8.00	.1583
9	5,487		9.00	.1985
10	5,418		10.00	.1960
11	17,915	251	11.00	.6481
12	27,502		12.00	.9950
13	8,321		13.00	.3010
14	677		14.00	.0245
15	277		15.00	.0100
16	257		16.00	.0093
17	204	258	17.00	.0074
18	133		18.00	.0048
19	241		19.00	.0087
20	435		20.00	.0157
21	892		21.00	.0323
22	764	264	22.00	.0276
23	337		23.00	.0122
24	185		24.00	.0067
25	155		25.00	.0056

I-131 Alone RUN I-4

Channel	Obs. Count Rate (c/m)	Elapsed Time, min	Norm. Channel	Norm. Count Rate
8	5,966	340	8.96	.1844
9	5,462		10.00	.1688
10	20,020		11.04	.6188
11	27,898		12.08	.8623
12	6,121		13.12	.1892
13	498	346	14.16	.0154
14	283		15.20	.0088
15	272		16.24	.0084
16	224		17.29	.0069
17	173		18.33	.0053
18	251	351	19.37	.0078
19	612		20.41	.0189
20	892		21.46	.0276
21	360		22.50	.0111
22	200		23.54	.0062
23	158		24.58	.0049
24	64			
25	14	360		

I-128 + I-131 RUN II-1

Channel	Obs. Count Rate (c/m)	Elapsed Time, min	Norm. Channel	Norm. Count Rate
8	4,739	6		
9	4,974	8		
10	7,360	9		
11	24,375	10	11.45	.9028
12	16,868	11	12.43	.6247
13	1,803	13	13.42	.0668
14	336	14	14.40	.0124
15	248	15	15.39	.0092
16	211	16	16.37	.0078
17	151	17	17.35	.0056
18	126	18	18.34	.0047
19	231	20	19.32	.0086
20	519	21	20.31	.0192
21	821	22		
22	464	23		
23	241	25		
24	175	26		
25	122	27		

I-128 + I-131 RUN II-2

Channel	Obs. Count Rate (c/m)	Elapsed Time, min	Norm. Channel	Norm. Count Rate
8	3,964	28		
9	4,855	29		
10	6,141	31		
11	20,285	32		
12	21,834	33	12.19	.8734
13	4,606	34	13.17	.1842
14	485	35	14.15	.0194
15	252	36	15.13	.0101
16	207	38	16.11	.0083
17	148	39	17.09	.0059
18	133	40	18.07	.0053
19	160	41	19.05	.0064
20	433	42	20.03	.0173
21	710	44		
22	613	45		
23	247	46		
24	154	47		
25	107	49		

I-131 Alone RUN II-3

Channel	Obs. Count Rate (c/m)	Elapsed Time, min	Norm. Channel	Norm. Count Rate
8	3,730	205	7.86	.1397
9	4,849		8.88	.1816
10	4,786		9.91	.1792
11	13,697		10.93	.5129
12	25,676		11.96	.9614
13	6,031	212	12.98	.2258
14	459		14.01	.0172
15	266		15.03	.0100
16	221		16.06	.0083
17	166		17.08	.0062
18	129		18.11	.0048
19	163	218	19.13	.0061
20	472		20.16	.0177
21	774		21.18	.0290
22	541		22.21	.0203
23	185		23.23	.0069
24	142		24.26	.0053
25	88	226	25.28	.0033

I-131 Alone RUN II-4

Channel	Obs. Count Rate (c/m)	Elapsed Time, min	Norm. Channel	Norm. Count Rate
8	5,340	302	8.96	.1873
9	5,070		10.02	.1778
10	18,999		11.07	.6663
11	26,191		12.12	.9185
12	4,980		13.17	.1747
13	379		14.22	.0133
14	294	309	15.28	.0103
15	216		16.33	.0076
16	79		17.38	.0028
17	148		18.43	.0052
18	305		19.48	.0107
19	662		20.54	.0232
20	783	316	21.59	.0274
21	351		22.64	.0123
22	176		23.69	.0062
23	147		24.74	.0051
24	58		25.80	.0020
25	11	321		

I-128 + I-131 RUN III-1

Channel	Obs. Count Rate (c/m)	Elapsed Time, min	Norm. Channel	Norm. Count Rate
8	4,140	6		
9	5,310	7		
10	6,514	9		
11	20,990	10		
12	27,107	11	12.07	.9681
13	8,201	13	13.05	.2930
14	475	14	14.04	.0170
15	253	15	15.03	.0090
16	218	16	16.02	.0078
17	197	17	17.00	.0070
18	153	18	17.99	.0055
19	129	20	18.98	.0046
20	372	22		
21	709	23		
22	607	24		
23	310	26		
24	162	27		
25	120	28		

I-128 + I-131 RUN III-2

Channel	Obs. Count Rate (c/m)	Elapsed Time, min	Norm. Channel	Norm. Count Rate
8	4,015	28		
9	5,215	29		
10	4,701	31		
11	12,371	32		
12	27,664	33	11.84	.9539
13	8,400	34	12.84	.2897
14	798	36	13.83	.0275
15	327	37	14.83	.0113
16	246	38	15.82	.0085
17	196	39	16.82	.0068
18	134	40	17.81	.0046
19	195	42	18.81	.0067
20	404	43		
21	788	44		
22	762	45		
23	280	46		
24	176	48		
25	118	50		

I-131 Alone RUN III-3

Channel	Obs. Count Rate (c/m)	Elapsed Time, min	Norm. Channel	Norm. Count Rate
8	4,767	168	8.23	.1605
9	4,955		9.34	.1668
10	9,219		10.39	.3104
11	27,118		11.43	.9130
12	17,545		12.48	.5907
13	1,364		13.53	.0459
14	352	176	14.58	.0119
15	239		15.63	.0080
16	261		16.68	.0088
17	165		17.73	.0056
18	143	180	18.77	.0048
19	380		19.82	.0128
20	790		20.87	.0266
21	714		21.92	.0240
22	302		22.97	.0102
23	156		24.02	.0053
24	102		25.07	.0034
25	24	187	26.11	.0008

I-131 Alone RUN III-4

Channel	Obs. Count Rate (c/m)	Elapsed Time, min	Norm. Channel	Norm. Count Rate
8	5,635	262	8.99	.1844
9	5,533		10.05	.1810
10	20,110		11.11	.6578
11	25,793		12.16	.8437
12	3,675		13.22	.1202
13	385		14.27	.0126
14	255	269	15.33	.0084
15	219		16.39	.0072
16	180		17.44	.0059
17	180		18.50	.0059
18	448		19.55	.0146
19	795		20.61	.0260
20	899	277	21.67	.0294
21	335		22.73	.0110
22	179		23.78	.0059
23	91		24.84	.0030
24	30		25.89	.0010
25	4	284	26.95	—

I-128 + I-131 RUN IV-1

Channel	Obs. Count Rate (c/m	Elapsed Time, min	Norm. Channel	Norm. Count Rate
8	4,352	6		
9	5,631	7		
10	5,015	8		
11	13,800	10		
12	29,690	11	11.75	.9897
13	13,138	12	12.73	.4379
14	1,011	13	13.70	.0337
15	341	15	14.67	.0114
16	253	16	15.64	.0084
17	234	17	16.61	.0078
18	185	18	17.58	.0062
19	139	19	18.55	.0046
20	348	20		
21	692	22		
22	841	23		
23	527	24		
24	228	25		
25	161	26		

I-128 + I-131   RUN IV-2

Channel	Obs. Count Rate (c/m)	Elapsed Time, min	Norm. Channel	Norm. Count Rate
8	4,151	28		
9	5,537	29		
10	6,033	30		
11	19,996	31		
12	29,424	33	11.99	.9974
13	8,576	34	12.93	.2907
14	656	35	13.87	.0222
15	332	36	14.80	.0113
16	250	37	15.74	.0085
17	205	38	16.68	.0069
18	170	40	17.62	.0058
19	182	41	18.56	.0062
20	393	42		
21	653	43		
22	883	45		
23	546	46		
24	224	47		
25	148	48		

I-131 Alone RUN IV-3

Channel	Obs. Count Rate (c/m)	Elapsed Time, min	Norm. Channel	Norm. Count Rate
8	5,869	124	8.72	.1845
9	5,181		9.77	.1629
10	16,950		10.83	.5329
11	31,339		11.89	.9852
12	10,288		12.94	.3234
13	650		14.00	.0204
14	329	132	15.05	.0103
15	259		16.11	.0082
16	208		17.16	.0071
17	167		18.22	.0052
18	189		19.27	.0059
19	472	137	20.30	.0148
20	951		21.38	.0299
21	621		22.44	.0195
22	214		23.50	.0067
23	190		24.55	.0060
24	15		25.61	.0005
25	2	145		

I-131 Alone RUN IV-4

Channel	Obs. Count Rate (c/m)	Elapsed Time, min	Norm. Channel	Norm. Count Rate
8	5,947	222	9.03	.1796
9	6,932		10.04	.2094
10	23,274		11.14	.7031
11	27,692		12.19	.8365
12	3,785		13.25	.1143
13	455		14.30	.0137
14	295	228	15.36	.0089
15	247		16.41	.0075
16	179		17.46	.0054
17	155		18.52	.0047
18	335	233	19.57	.0101
19	732		20.63	.0221
20	60		21.68	.0018
21	406		22.73	.0123
22	170		23.79	.0051
23	177		24.84	.0053
24	59		25.90	.0018
25	14	241		

I-128 - I-131 DIFFERENCES

Run No.	Norm. Channel	Difference	Weighted Difference*
I-1	14.59	+38.4	+ 37.4
	15.67	+16.0	+ 5.2
	16.75	-16.0	- 0.1
II-1	14.40	+ 2.7	+ 2.3
	15.39	+10.8	+ 6.2
	16.37	- 5.4	- 0.2
III-1	14.04	0.0	0.0
	15.03	-16.8	-15.0
	16.02	-14.0	- 1.7
IV-1	14.67	+12.0	+ 11.9
	15.64	0.0	0.0
	16.61	+ 3.0	0.0
I-2	14.63	+18.6	+ 18.4
	15.62	+31.0	+ 0.6
	16.61	+27.9	+ 0.1
II-2	14.15	+105.0	+ 68.2
	15.13	+17.5	+ 10.2
	16.11	+ 2.5	0.0
III-2	14.83	+31.9	+ 31.6
	15.82	+ 2.9	+ 0.1
	16.82	-17.4	0.0
IV-2	14.80	+29.5	+ 29.3
	15.74	0.0	0.0
	16.68	-20.6	- 0.1

*Weighted differences not decay corrected.

APPENDIX II

I-129 DETERMINATION

I-129 + I-131 Run 1

Channel	Obs. Count Rate (c/m)	Elapsed Time, min	Norm. Channel	Norm. Count Rate
14	11,269	37	15.00	.1483
15	18,311		16.11	.2409
16	58,794		17.23	.7736
17	74,557		18.35	.9810
18	45,405	42	19.46	.5974
19	7,379		20.58	.0971
20	2,056		21.70	.0271
21	1,387		22.81	.0183
22	1,096	47	23.93	.0144
23	1,010		25.05	.0133
24	849		26.17	.0112
25	692		27.28	.0091
26	879	52	28.40	.0116
27	853		29.52	.0112
28	1,586		30.63	.0209
29	2,368		31.75	.0312
30	2,155	57	32.87	.0284
31	1,088		33.98	.0143
32	665		35.10	.0088
33	629		36.22	.0083
34	476		37.34	.0063
35	156	62	38.45	.0021

I-129 + I-131 RUN 2

Channel	Obs. Count Rate (c/m)	Elapsed Time, min	Norm. Channel	Norm. Count Rate
14	12,117	65	15.46	.1478
15	30,602	67	16.53	.3732
16	67,525		17.60	.8235
17	68,905		18.67	.8403
18	23,946		19.74	.2920
19	4,638		20.81	.0566
20	1,844		21.88	.0225
21	1,310	74	22.95	.0160
22	1,042		24.02	.0127
23	943		25.09	.0115
24	834	77	26.16	.0102
25	701		27.23	.0085
26	722		28.30	.0088
27	816		29.37	.0100
28	1,491	82	30.44	.0182
29	2,293		31.51	.0280
30	2,430		32.58	.0296
31	1,328		33.65	.0162
32	670	87	34.72	.0082
33	610	88	35.79	.0074
34	527		36.86	.0064
35	287		37.93	.0035

I-131 Alone RUN 3

Channel	Obs. Count Rate (c/m)	Elapsed Time, min	Norm. Channel	Norm. Count Rate
16	13,357	5160	16.00	.2671
17	33,357		17.00	.6671
18	49,615		18.00	.9923
19	36,880		19.00	.7376
20	9,834		20.00	.1967
21	1,544		21.00	.0309
22	906		22.00	.0181
23	714		23.00	.0143
24	621		24.00	.0124
25	606		25.00	.0121
26	465		26.00	.0093
27	382		27.00	.0076
28	365		28.00	.0073
29	498		29.00	.0100
30	847		30.00	.0169
31	1,143		31.00	.0229
32	1,651		32.00	.0330
33	1,333		33.00	.0267
34	732		34.00	.0146
35	420		35.00	.0084
36	397		36.00	.0079
37	293		37.00	.0059